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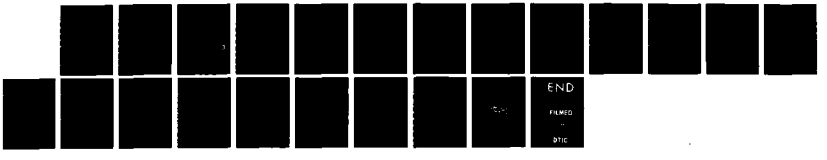

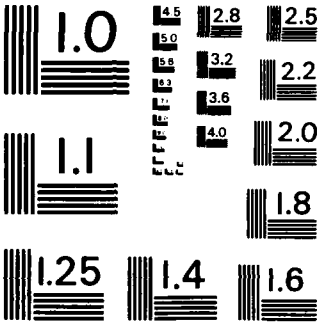

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A Tunneling Microscope for Operation in Air or Fluids

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Abstract

A tunneling microscope that is a hybrid between IBM Zurich designs and squeezable tunnel junctions has been operated in air, oil, and liquid nitrogen. Key design goals were 1) maximum rigidity and 2) minimum thermal drift. Images of individual atoms in a close packed layer have been obtained under liquid nitrogen.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A tunneling microscope that is a hybrid between IBM Zurich designs and squeezable tunnel junctions has been operated in air, oil, and liquid nitrogen. Key design goals were 1) maximum rigidity and 2) minimum thermal drift. Images of individual atoms in a close packed layer have been obtained under liquid nitrogen.

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Introduction

Tunneling microscopy is evolving into a powerful tool for surface topography and analysis. Milestones in this evolution have included profiling grating surfaces[1,2], observing steps one atom high[3,4,5], detailing the atomic positions in semiconductor reconstructions[6,7], demonstrating small scale variations in the superconducting energy gap at the surface of a thin film[8], observing single atoms in a close-packed layer[9], and spectroscopic imaging[10].

One of the advantages of tunneling microscopy is that it is not limited to studying vacuum-solid interfaces. It can also be used to study gas-solid and liquid-solid interfaces: the interfaces of most importance in catalysis, corrosion, lubrication and biology.

The design reported here has been used for operation in air, oil, and liquid nitrogen and should be useful in other gases and liquids. It is a hybrid between IBM Zurich designs[3-6] and squeezable electron tunneling junctions[11-14].

Design

a) X-Y TRANSLATORS

Two designs for X-Y translators are shown in fig. 1. Design A is the original design. Design B is used currently. Both X-Y translators are cut from a block of Channel 5400 piezoelectric material.[15] Both use 1.5mm diameter spacers approximately 1.2mm in height to locate the Z-translator. The collet, made from a 2-56 brass screw, holds the scanning tip. The major design differences are the mounting configurations. Translator A is fastened to the translator support (fig. 3) using a machined ceramic (Corning Macor) ring while design B is bolted directly to the support. Design B is superior because of its simplicity, increased rigidity, and improved sample alignment. The signs along the arms of the translators signify their poling direction.

The x motion is generated by applying a voltage so that the electric field is in the direction of poling for one arm and opposite to the direction of poling for the arm across from it. Thus, one arm expands and one contracts, moving the collet and tip to one side relative to the spacers, while maintaining the total distance between the spacers the same. The motion is of order 4.3 Å/V at room temperature and 3.3 Å/V at liquid nitrogen temperature.

The y motion is generated in the same way as the x motion for design A. For design B the voltage is applied to the one remaining y translation arm which moves the collet and tip by bending the x translation arms. The calibrations for the y axes are roughly the same as for the x axes.

The most direct way to calibrate the translator is in terms of an atomic image of a known structure. A piezoelectric arm may also be calibrated by attaching a mirror and including it in 2 Michelson interferometer, or by attaching a capacitor plate and including it in a capacitance bridge (e.g. a Type 1615 General Radio Capacitance Bridge). These methods are in reasonable agreement with data supplied by the manufacturer.

b) Z translator

The Z translator is a half circular disk made of Channel 5800 piezoelectric material. It is 3 cm in diameter and composed of two layers each 1mm thick glued together with epoxy. We have also experimented with layers 0.5mm thick and, more recently, 1.5mm thick. The thicker the layers the greater the rigidity, but the smaller the range since the motion is proportional to one over the square of the thickness. For 1 mm thick layers the motion is roughly 60 Å/V at room temperature and 36 Å/V at liquid nitrogen temperature based on the manufacturer's values for d_{31}, g_{31} and ε_{33} followed by extrapolation to 77K using another manufacturer's data[16] for similar materials[17].

c) Translator Assembly

Fig. 2 shows a side and sectional view of the translator assembly. The Z-translator is shown in the operating position with the sample mounted on the bottom surface. Typically the sample is mounted with fast drying glue (Duco) or varnish (GE7031) on a glass cover slip which is glued to the bimorph. Section A-A shows the collet with the scanning tip, drill blank spacer and .5UNM threaded wire. The collet is made with a press fit on the .5mm diameter PtIr tip. The press fit allows for smooth advancement of the tip position as the threaded wire and drill blank are advanced. When the threaded wire and drill blank are retracted the tip will retain its position. The drill blank is used to isolate the tip from the rotation of the threaded wire.

d) Translator Support

The function of the translator support shown in Figure 3 is to hold rigidly the X-Y translator while allowing the scanning tip to be advanced toward the sample. The drawing shows the rotatable knurled disc raised to engage the .5UNM threaded wire. In this position the point can be dependably advanced to within approximately .1um of the sample. Once the tip height is adjusted within the operating range of the Z-translator the threaded wire is retracted and the knurled disc is lowered to increase thermal and vibrational stability. The translator support can be hung (fig. 5) or allowed to rest on its base (fig. 4). When it rests on its base the Z translator will stay in place on the X-Y translator just by gravity. When it is hung, the Z translator must be more firmly attached to keep it from slipping if the translator support bumps the side of the container. At first we used glue (duco) at the feet, but more recently we have used a spring clip fastened to the translator support and pressing on top of the bimorph as shown in Fig. 3(b)(spring clip can also make the electrical connection to the top of the bimorph. The rear foot is used as the bottom electrical connection).

The electrical leads are fastened to a Macor ring near the top of the translator support with 2-56 screws.

e) Air Microscope

Fig. 4 is a simplified drawing of the vibration isolation apparatus used in the air microscope at UCSB. Two independent vibration isolation mechanisms are employed. An air table (NRC model GS-34) supports the entire system while a three spring suspension scheme is used internally. An oil damper is used on the three spring internal suspension. The rod to the damping fins is shown below the spring-supported platform.

The bell jar provides roughly 30dB of acoustic attenuation from 10 Hz to 20kHz. The inner shield (a coffee can) provides electrical screening and shuts out light.

Not shown is approximately 150kg of lead that was hung below the air table to lower the center of gravity and thus provide a more stable equilibrium.

f) LN_2 Microscope

Fig. 5 is a sectional view of the LN_2 microscope at the University of Virginia. The LN_2 microscope also uses two independent vibration isolation systems. Instead of an air table, the support frame is suspended from the ceiling using latex tubing, which has proven reliable and considerably less troublesome than the air table. The secondary isolation is a steel spring attached directly to the translator support.

A mechanical pump is used to evacuate the bell jar & inner dewar chamber before the translator support is lowered in order to remove as much water vapor as possible since it will condense on the sample during the gradual cooling of the translator support as it is lowered into the LN_2. This evacuation is performed with LN_2 in the inner dewar. Boiling chips help keep the LN_2 from bumping, but do not totally eliminate the problem. A valve between the dewar and bell jar would be a helpful addition.

A two r.p.m. reversible motor is used to lower and raise the translator support with a string. To engage the secondary vibration isolation the translator support is lowered into the LN_2 until the string is slack. When both dewars

are filled with LN_2 it is possible to have 16 hours of operating time before refilling. Both dewars can be filled with the translator support either raised or lowered.

g) Electronics

Figure 6 shows a block diagram of the electronics. The only major component that needed to be custom built was the logarithmic integrating error amplifier. It uses an Analog Devices 755P logarithmic amplifier, an OP27 for integration and a LH0002 for output. The tip to sample current was monitored by measuring the voltage across a 1MΩ series resistor with a PAR113 preamplifier operated at a gain of 100. The high voltage amplifiers for driving the piezoelectric translators were homebuilt with a TCG238 output transistor, but alternate ones could be obtained commercially (from Kepco).

Experimental Results

a) In Air

Figure 7a shows an evaporated gold film imaged in air with the microscope shown in Figure 4. This microscope has also been used to image organic conducting polymers on evaporated gold films, highly oriented pyrolytic graphite (HOPG), carbon coated transmission electron microscope grids and 2H-$TaSe_2$. Though atomic height steps have been observed, individual atoms have not. Some rather mysterious periodic features that seem to imply sub-angstrom lateral resolution on HOPG have been obtained, but we neither understand the images nor their implications at present.

b) In Oil

Figure 7b shows an evaporated gold film imaged with the microscope shown in Figure 4 immersed in paraffin oil. We have not done much work under oil because of the difficulty in either cleaning the microscope after use or alternately trying to mount samples while it is covered with oil.

c) In Liquid Nitrogen

Figure 8 shows atoms on a cleaved surface of 2H-$TaSe_2$. At 77K this material exhibits a commensurate charge-density wave, but it is not sufficiently strong to dominate the atoms imaged with the tunneling microscope[18]. This microscope has also been used to image 1T-TaS_2 and in this case charge-density waves were dominant due in part to the much larger charge-density wave amplitude[18].

Only with the microscope immersed in liquid nitrogen have we been able to image individual atoms. Advantages of liquid nitrogen that may play a role are: 1) it provides a relatively inert environment, 2) it decreases the observed z axis noise, 3) it provides damping of piezoelectric resonances and 4) it decreases thermal drifts.

Discussion

a) Vibration Isolation

The most critical vibration isolation problem for these microscopes is vertical vibration. The microscopes are relatively immune to horizontal vibrations since both the x-y translator and the z translator are relatively rigid in the horizontal direction and they are coupled by short stocky spacers.

The vertical vibration amplitude as measured with a geophone, is of order 1μm @ 10 Hz at both sites. This is attenuated by a factor of roughly 10 by the vibration isolation.

If the microscopes were perfectly rigid in the vertical direction the tip and sample would move together and there would be no net motion between them. The departure from this perfect situation can be estimated from a simple formula. The tip to sample distance, S, will change by

$$\Delta S = C(f/f_R)^2 A \text{ for } f \ll f_R$$

where C is a constant of order unity that depends on the damping, f is the frequency of the vibration, A is the amplitude of the vibration, and f_R is the resonant frequency of the z translator, since it is much less rigid than the x-y translator for vertical vibrations.

We found f_R = 11kHz by applying an ac voltage of 0.1V rms and listening for the response as the frequency was changed. A much louder sound could be heard clearly as the frequency goes through the resonant frequency. Thus 1.5×10^{-3} Å, which is small enough to be unobservable at our current electrical noise level.

Though the immunity to vibrations decreases as the square of the vibrational frequency, the attenuation of high frequency vibrations by the double vibration attenuation system coupled with the lower amplitude of these vibrations at both sites make them less troublesome than the low frequency vibrations.

b) Thermal Drifts

In the Z direction the mismatch in thermal expansion coefficients between the spacers and the collet plus tip is of order $5 \times 10^{-6} / {}^{\circ}C$ over a length of 1.2mm. Thus thermal drifts of order 60 Å/°C can be expected. In the x-y direction the mismatch between the z translator and the x-y translator is $<10^{-6}/{}^{\circ}C$ over a length of 1.3cm. Thus thermal drifts of less than 130Å/°C can be expected. We are prevented from making unambiguous measurements of thermal drift by piezoelectric creep, which is a more significant problem.

The thermal drifts are, however, troublesome in one respect. Since the liquid nitrogen microscope is coarse adjusted at room-temperature and then lowered it is important that thermal drift not take the z translator outside its adjustment range. In practice the voltage on the z translator changes by of order 200V during cooldown implying a change in distance of order 1μ, which is consistent with the estimate above. Sometimes, however, the voltage changes by more than this-reaching the limit we are willing to apply $(\sim 400V)$ and preventing us from taking data. We associate this behavior with Z translators which have been partially depoled since their thermal expansion coefficient depends on poling. We are presently working on a design to allow us to do coarse adjustment under the liquid.

c) Piezoelectric Creep

Piezoelectric creep is still a problem for these microscopes despite the fact that we selected piezoelectric materials specifically for low creep. It is perhaps most dramatic in the z axis of the room temperature microscope. For example, if the voltage to bring the sample to within tunneling distance of the tip starts at 150V it will typically decrease to of order 100V over the period of 15 minutes. If this were interpreted as a distance change it would be 6,000Å! Thus we are prevented from taking data until this creep subsides. Fortunately it decreases to of order 10 Å/min after 15 min., 2nd to 4Å/min. after 60 minutes. In liquid nitrogen the creep is not as fast, but it persists longer.

Summary

1) A tunneling microscope that is a hybrid between IBM Zurich designs and squeezable electron tunneling junctions can be operated in air and submerged in non-conducting fluids, including LN_2.
2) Resolution sufficient to image individual atoms in a close-packed, unreconstructed layer is obtainable with submersion in liquid nitrogen. This implies lateral resolution better than 3.4Å. The best vertical resolution was of order 0.1Å.
3. Surface topography with lateral resolution of order 10Å and vertical resolution of order 1Å can be done in air or under oil.

Acknowledgements

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References

1. R. Young, Phys. Today 24, 42 (1971)
2. R. Young, J. Ward, and F. Scire, Rev. Sci Instrum 47, 1303 (1976)
3. G. Binnig, H. Rohrer, Ch. Gerber, & E. Weibel, Phys. Rev. Lett. 49, 57 (1982)
4. G. Binnig & H. Rohrer, Surf. Sci 126, 236 (1983)
5. G.K. Binnig, H. Rohrer, Ch. Gerber & E. Stoll, Surf. Sci 144 321 (1984)
6. G. Binnig, H. Rohrer, Ch. Gerber & E. Weibel, Phys. Rev. Lett 50, 120 (1983)
7. J.A. Golovchenko, Bull. Am. Phys. Soc 30, 251 (1985)
8. S.A. Elrod, A.L. de Lozanne, and C.R. Quate, Appl. Phys. Lett 45, 1240 (1984)
9. C. Quate, Bull. Am. Phys. Soc 45, 251 (1985)
10. G. Binnig, Bull Am. Phys. Soc 30, 251 (1985)
11. J. Moreland, S. Alexander, M. Cox, R. Sonnenfeld, and P.K. Hansma, Appl. Phys. Lett 43, 387 (1983)
12. J. Moreland & P.K. Hansma, Rev. Sci Instrum 55, 399 (1984)
13. J. Moreland, J. Drucker, P.K. Hansma, J.P. Kotthaus, A. Adams & R. Kvaas, Appl. Phys. Lett 45, 104 (1984)
14. R. Sonnenfeld, J. Moreland, P.K. Hansma, A. Adams and R. Kvaas, J. Appl. Phys. 58, 392 (1985).
15. Channel Industries, Santa Barbara, CA.
16. "Piezolectric Technology Data for Designers", Vernitron, Bedford, Ohio.
17. C5400 is similar to PZT4; C5800 is similar to PZT8 according to M. Cullen of Channel Industries.
18. R.V. Coleman, B. Drake, P.K. Hansma & G. Slough, Phys. Rev. Lett 55, 394 (1985).

Figure Captions

1) The x-y translators move the collet (and the tip it holds) relative to the spacers that support the Z translator.

2) A side view shows the Z translator supported above the x-y translator by the spacers. The Z translator warps downward with applied voltage to keep the tip to sample distance constant.

3)(a) The translator support is machined from aluminum and then anodized.

(b) Photograph of microscope showing bimorph being installed.

4) The air microscope at UCSB is acoustically isolated with a bell jar and vibrationally isolated with an air table and springs.

5) The LN_2 microscope at UVa is acoustically isolated with the bell jar and vibrationally isolated with rubber tubing and a spring.

6. A block diagram of the electronics.

7. Evaporated gold films on glass cover glasses imaged in air and under oil with the microscope shown in figure 4. The scales show the x and y calibrations. The z calibrations for both micrographs are the same. The height of the taller structures in both micrographs is $\approx$50Å.

8. 2H-$TaSe_2$ atoms imaged under LN_2 with the microscope shown in figure 5. The scales show the x & y calibrations. The height of the bumps due to the atoms is $\approx$0.5Å.

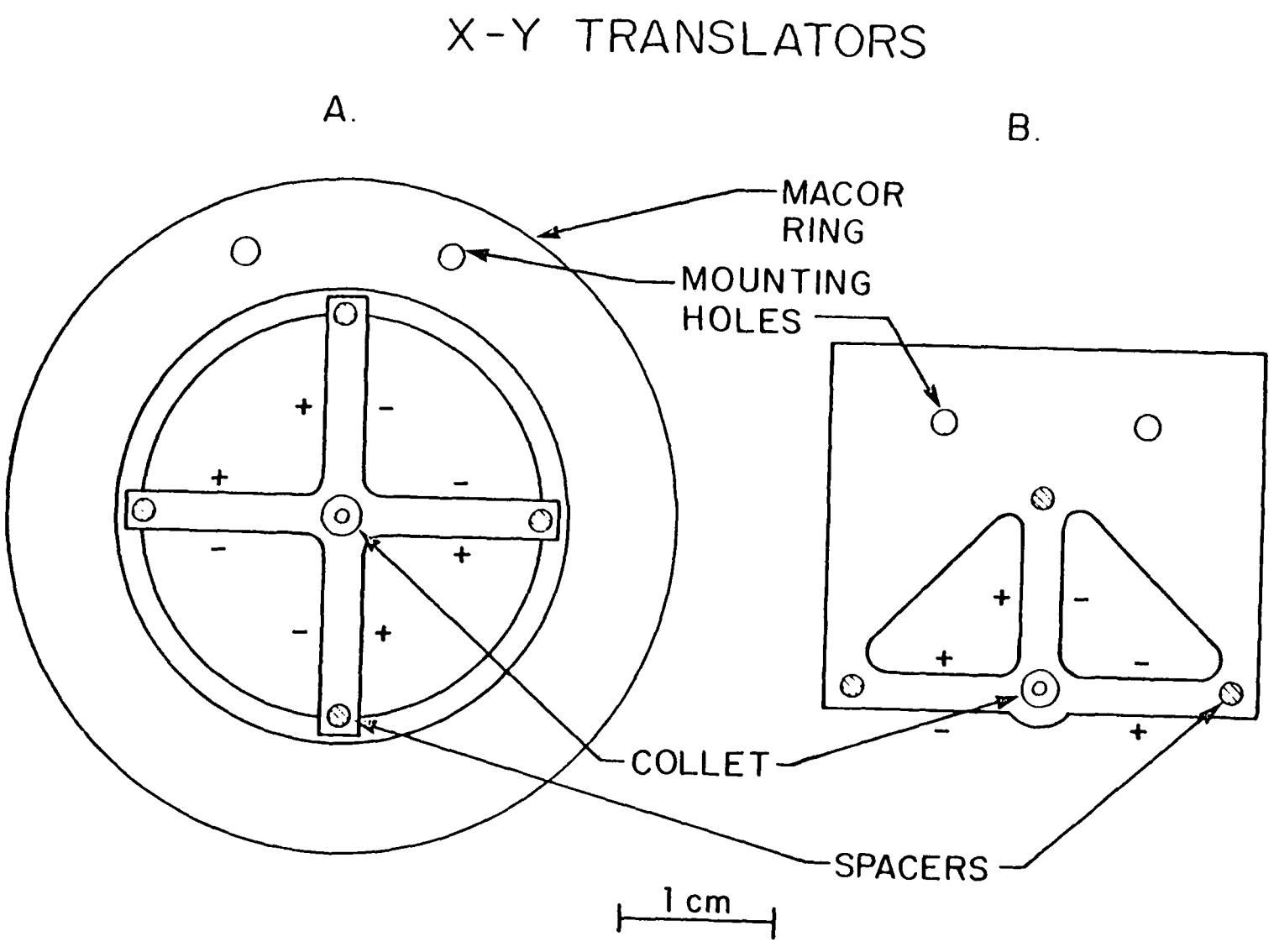

X-Y TRANSLATORS
A.
B.
MACOR RING
MOUNTING HOLES
COLLET
SPACERS
1 cm

FIG. 1

PIEZOELECTRIC TRANSLATORS

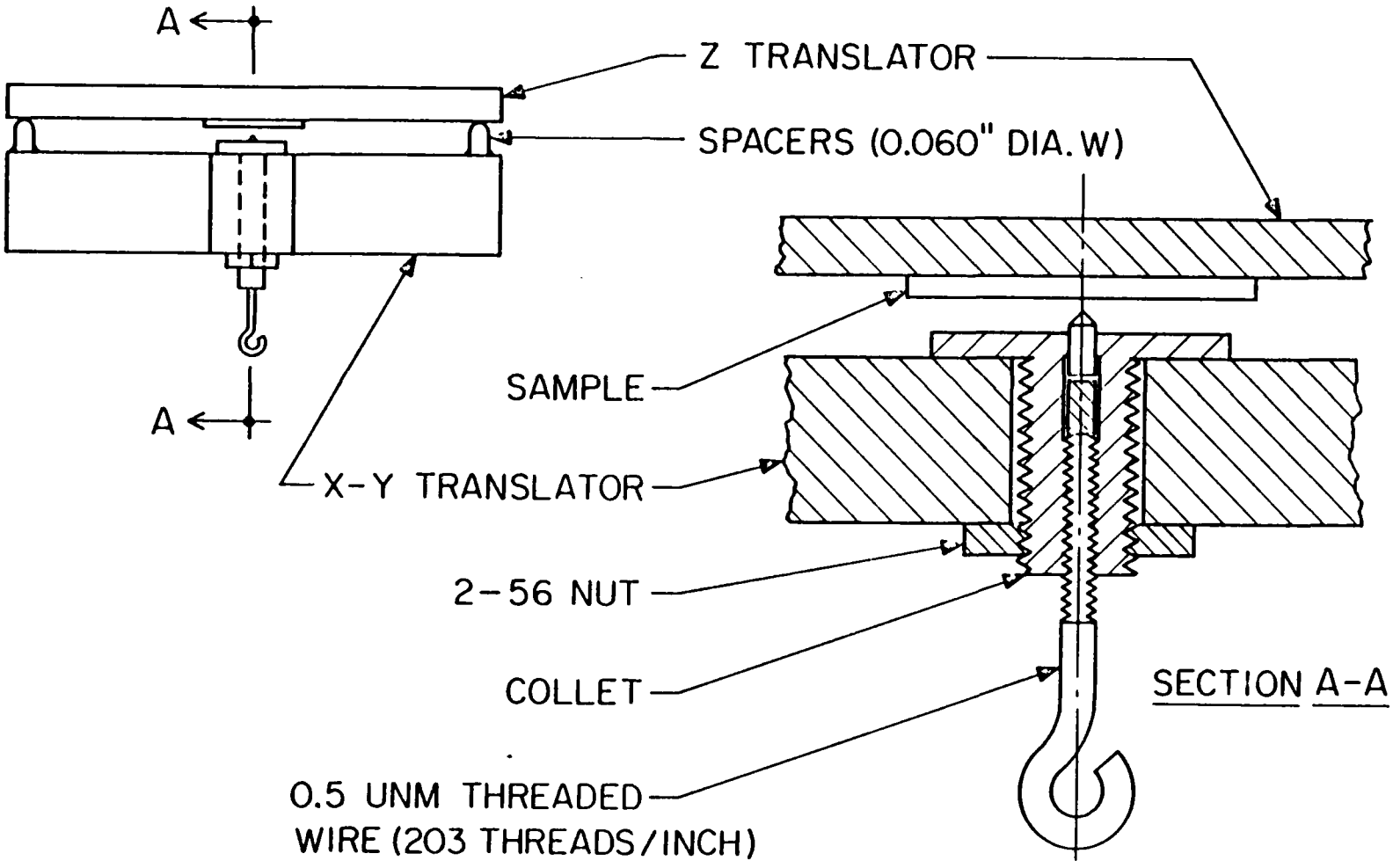


FIG. 2

TRANSLATOR SUPPORT

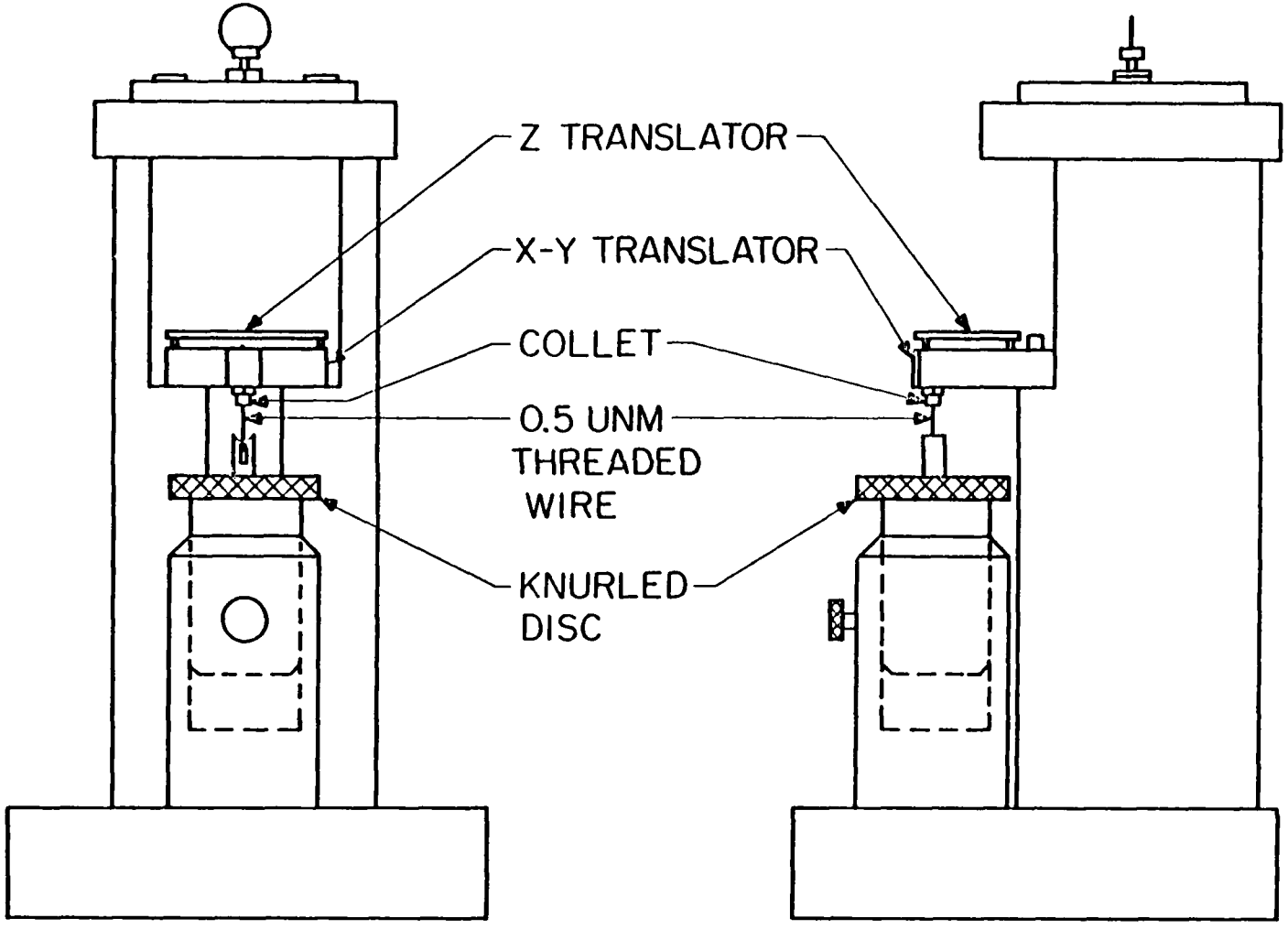


FIG. 3(A)

AIR MICROSCOPE

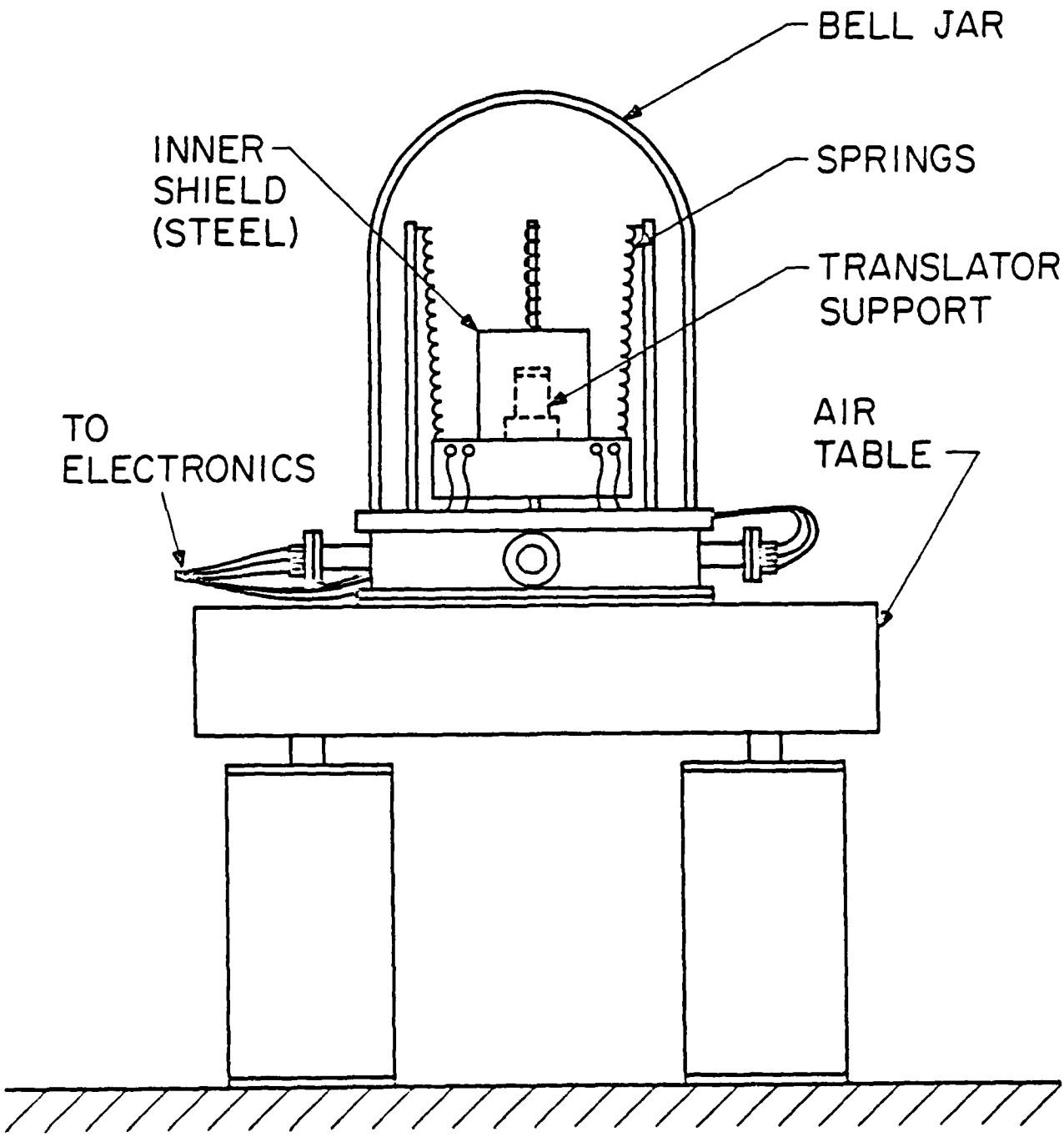


FIG. 4

LN_2 MICROSCOPE

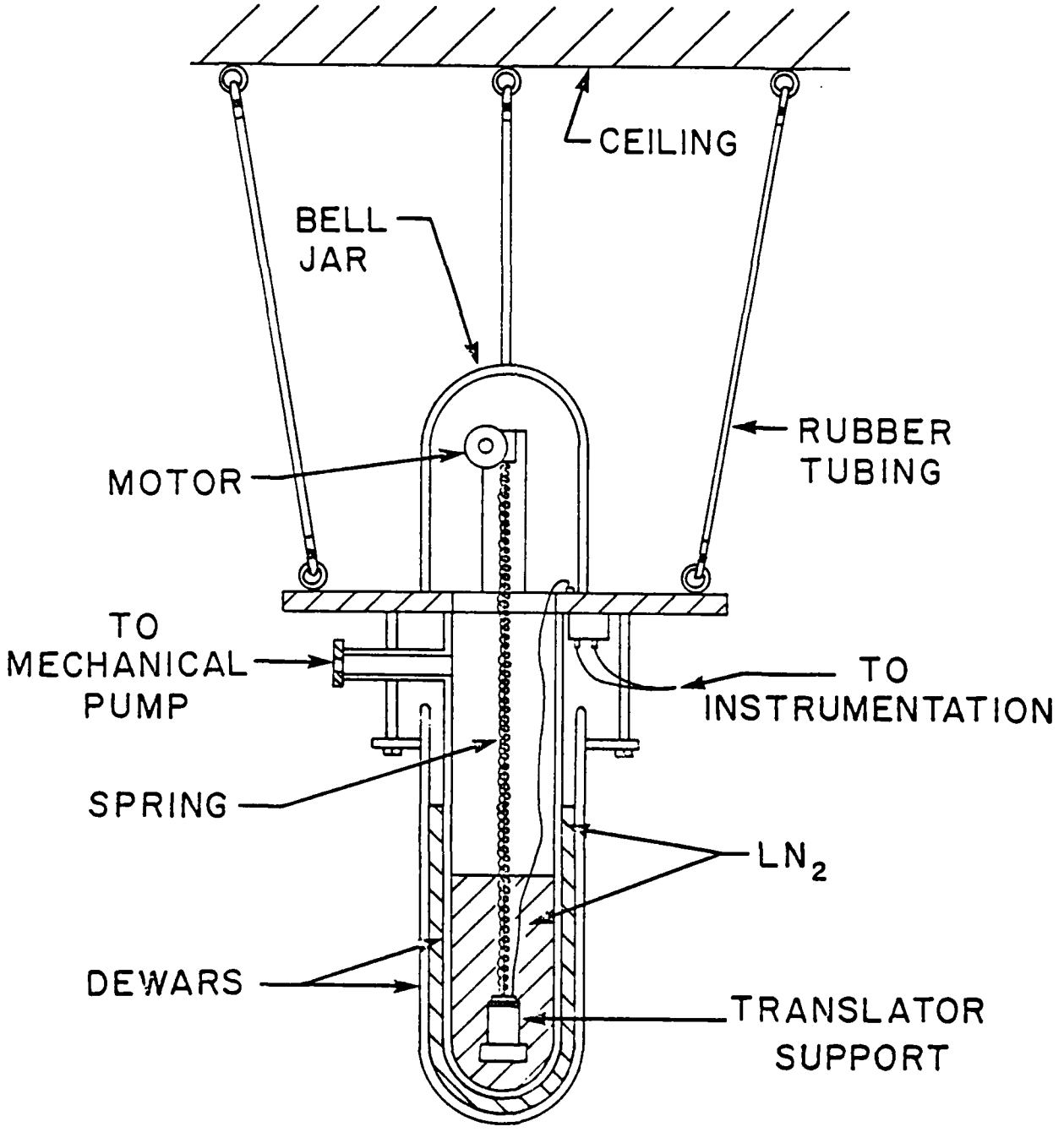
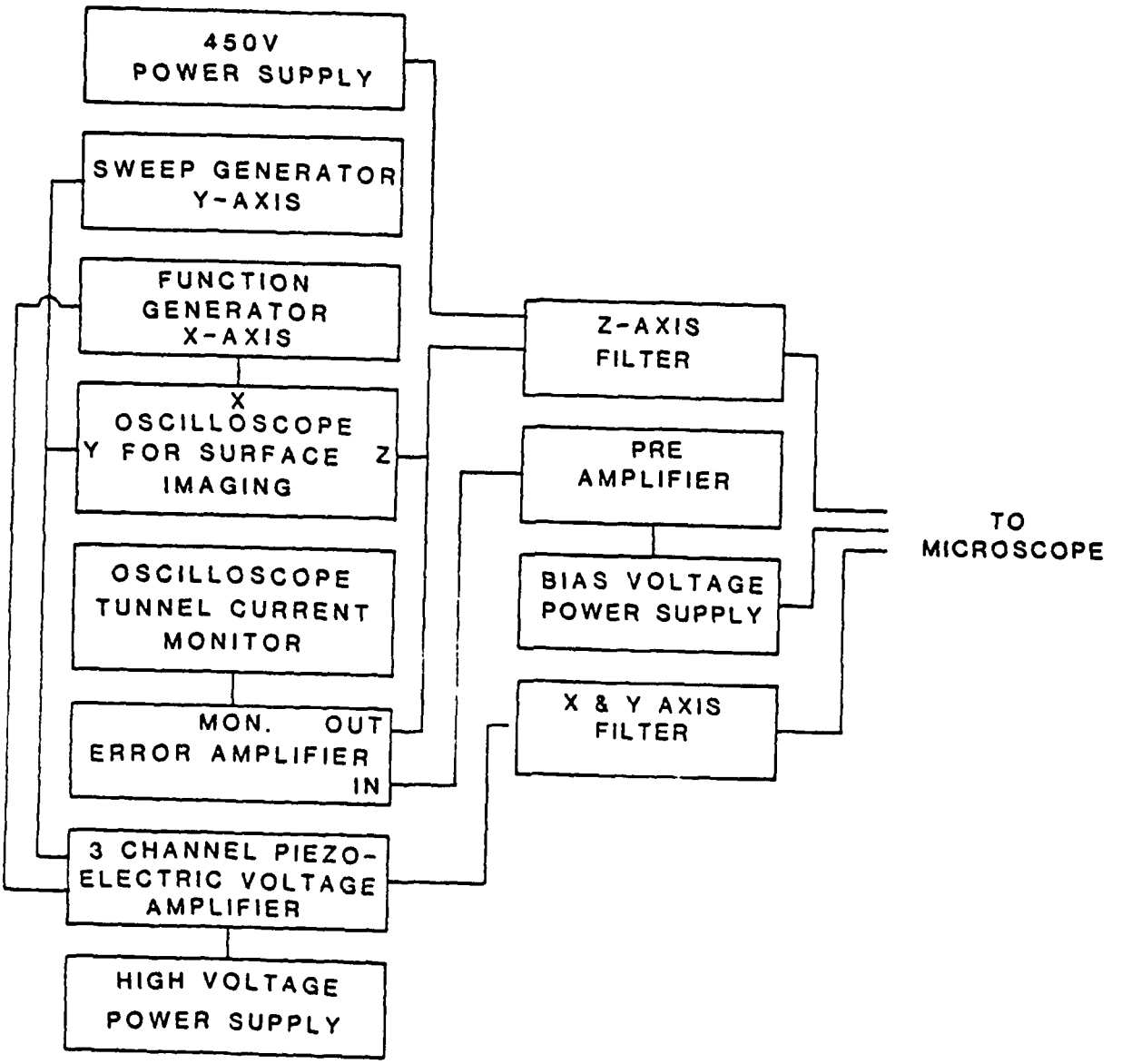


FIG. 5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POWER SUPPLY
SWEEP GENERATOR
Y-AXIS
FUNCTION
GENERATOR
X-AXIS
X
OSCILLOSCOPE
Y FOR SURFACE Z
IMAGING
OSCILLOSCOPE
TUNNEL CURRENT
MONITOR
MON. OUT
ERROR AMPLIFIER
IN
3 CHANNEL PIEZO-
ELECTRIC VOLTAGE
AMPLIFIER
HIGH VOLTAGE
POWER SUPPLY
Z-AXIS
FILTER
PRE
AMPLIFIER
BIAS VOLTAGE
POWER SUPPLY
X & Y AXIS
FILTER
TO
MICROSCOPE

FIG. 6

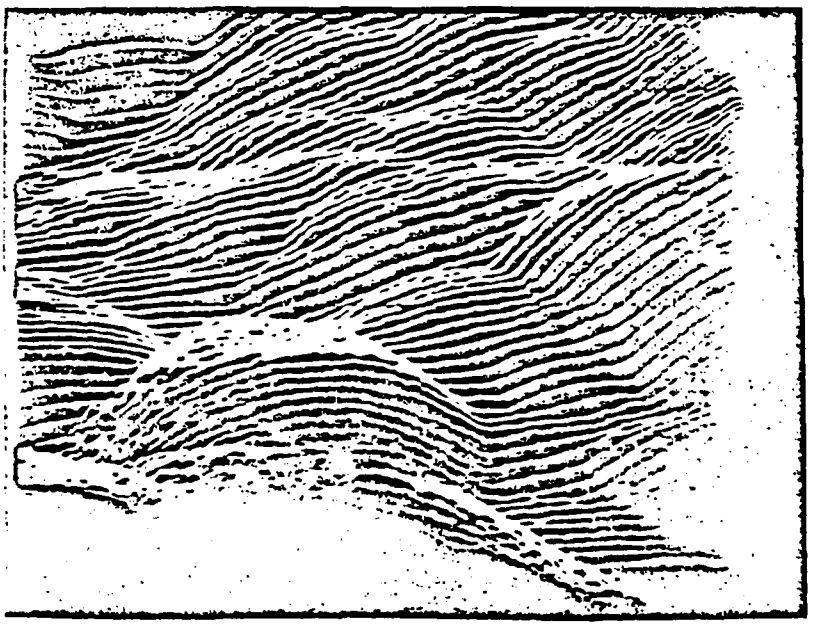
1000 Å Au FILM
IMAGED IN AIR

10 nm

10 nm

(a)

1000 Å Au FILM
IMAGED UNDER PARAFFIN OIL



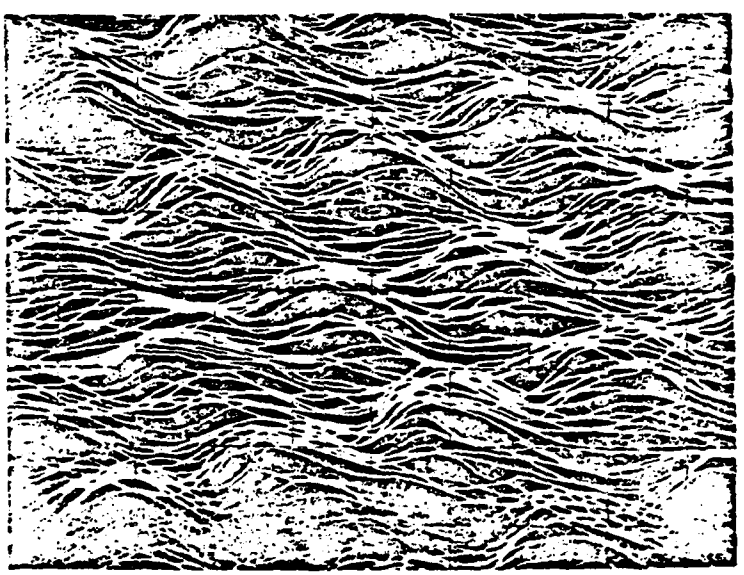
10 nm

10 nm

(b)

FIG. 7

2H-$TaSe_2$



3 Å

3 Å

FIG. 8

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